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TARDEC

Field Demonstration For Biodegradable Military Multipurpose Grease

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by

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13. ABSTRACT (Maximum 200 words) Soils and ground water at many military facilities throughout the United States have been contaminated with petroleum based products, such as engine oils, greases, hydraulic fluids, and fuels, often as a consequence of leakage, accidental spills, or equipment breakdown during active use or storage. The generation of hazardous waste by Petroleum, Oils, and Lubricant (POL) products results in both short and long term liability in terms of costs, environmental damage, and mission performance. For this reason, the military is currently soliciting a new technology that can be used to minimize the waste stream of these products. As a part of this program, a biodegradable lubricating grease (BLG) was successfully developed to replace the conventional petroleum based military automotive greases. This product has been fully evaluated both in the laboratory and under field operational environments. It provides an excellent biodegradability and performance that can meet the military global operational requirements. This paper presents the results of development of military biodegradable greases, laboratory biodegradation tests, and field demonstrations including bioremediation tests.

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1. BACKGROUND

Environmental safety and compliance has recently become the most significant worldwide issue. Over the past decades, many military installations throughout the United States have been contaminated with petroleum and related fuels, lubricants and associated products, such as lubricating oils, greases, hydraulic fluids, aircraft and automotive fuels, and those fuel used for fixed installations [1]. The environmental threat or damage to soils, surface water, and underground water were often caused by leaking containers, accidental spills, or equipment breakdown during active use or storage of these materials. The generation of the potentially hazardous wastes by Petroleum, Oil, and Lubricant (POL) not only cause both short and long term liability with respect to environmental damage, but can result in deteriorated mission performance and high cleanup costs. Currently, the Resource Conservation and Recovery Act (RCRA)[2] and the DoD Hazardous Waste Minimization (HAZMIN) Policy mandate that all DoD installations must reduce the quantity or volume and toxicity of hazardous waste generated by POL products wherever economically practicable and environmentally necessary. To achieve the HAZMIN goals, research and development efforts are being directed to develop new or improved HAZMIN techniques and processes.

In a survey, the Department of Defense (DoD) annually procures and consumes a large amount of POL in various applications. This results in a significant volume of used and off-specification products generated at DoD installations. To reduce this waste stream, recycling and refining technologies were recently introduced into the military community. Many lubricating oils are being recycled or re-refined, or can be burned for their energy value. Unlike oils, solid and/or semi-solid lubricants such as lubricating greases can not be considered for recycling due to difficulty in their liquefaction. Because of this, most military greases do not meet the DoD HAZMIN goals. For this reason, many military users have shown an increased interests in the development of new, biodegradable greases which appears to be very promising for addressing this problem from the front end [3,4,5].

In response to the demand of military biodegradable lubricating greases (BLGs), a program was initiated to develop a multipurpose BLG product which can rapidly biodegrade and will be less toxic to environment while, at the same time, providing satisfactory field performance in military applications. As a plan, the BLG product was originally designed as a potential replacement for the military automotive greases, MIL-G-10924G, Grease, Automotive and Artillery (GAA) [6]. This program has involved the following three phases.

Phase I: Develop target requirements for BLG

Phase II : Formulate and evaluate experimental BLG

Phase III: Conduct field demonstration to validate candidate BLG

During a period of 1994-1996, the target requirements of BLG were developed, and designed to satisfy both multipurpose applications and environmentally acceptability requirements. As a follow-up action, an experimental BLG product has been completely formulated, its physical/chemical properties were evaluated under laboratory environments. This BLG product is fully compatible with military GAA greases meeting MIL-PRF-10924G, and was formulated with advanced lithium complex thickener, synthetic oils, and additives. No toxic materials were included in this formulation. The laboratory test results showed this experimental BLG met all target requirements including a new biodegradation property. Because this product never had been used for military applications, its performance in field environment was viewed as an essential follow-on effort.

2. DEVELOPMENT OF CANDIDATE BIODEGRADABLE GREASES

The military has in the past been using a National Lubricating Grease Institute (NLGI) Number 2 consistency grease covered by military specification, MIL-G-10924, as the standard grease for all military vehicles and artillery and ground equipment operated worldwide. This grease, no obsolete, was originally designed for used in extreme field environments and for multipurpose applications. To meet these requirements, the formulation of the grease consisted of a blend of 6 cSt Polyalphaolefin (PAO) synthetic base stock, and a high viscosity mineral oil thickened with an advanced lithium complex thickener system. This grease has a wide operational capability (-54 to 180 °C), excellent water and storage stability, good shear and oxidation stability, good antiwear and load carrying capacity, and saltwater corrosion properties. One of disadvantages is its low biodegradability. For this reason, the MIL-G-10924 grease is not considered as a biodegradable product.

To improve its environmental properties, new target requirements were developed based on the requirements of MIL-G-10924 specification and what is believed to be achievable with current biodegradable grease formulation technology. These target requirements are listed in Table 2. In these requirements, the American Society for Testing and Materials (ASTM) D5864 method was adopted for biodegradation tests with its readily-biodegradable criteria. For the toxicity test, the Organization for Economic Co-Operation and Development (OECD) acute toxicity method was also adopted for this preliminary target requirements,

Effort on the development of experimental biodegradable grease was directed toward comparing the target requirements of new proposed biodegradable MIL-PRF-10924H grease to the performance potential of available biodegradable materials and environmentally acceptable additive technology. To minimize a compatibility problem, a formulation guideline was developed based on a lithium complex technology with advanced additive formulation used in the MIL-G-10924 greases. Initially, three experimental BLG products were formulated with a grease manufacturer. First, two experimental greases (BLG-1,2) were reformulated based on non-biodegradable MIL-G-10924F grease formulation, while BLG-3 was formulated with a low viscosity blended ester oils. The MIL-G-10924F grease was originally formulated with 65% by weight of polyalpaolefin (PAO) oil, 10% of petroleum oil, 15% of lithium-complex thickener, and 10% of the additives. In an earlier study, it was found that this formulation has a low biodegradability due to the non-biodegradable base oils. To improve its biodegradability, this formulation was modified using a blend technique that is often used with success in the formulation of the conventional petroleum-based greases. A rapeseed oil was selected as a biodegradability improver based on its high biodegradation performance. Initially, an experimental BLG-1 was prepared adding 10 percent of rapeseed oil into MIL-G-10924F, while BLG-2 were reformulated by adding 20 percent of rapeseed oil while deleting the petroleum oil and some PAO oil from this formulation. In another formulation, BLG-3 was a fresh formulation with a blend of polyol ester, diester and PAO. Table 1 provides the composition of BLG-1, BLG-2, and BLG-3 as compared to the baseline MIL-G-10924F grease.

Table. 1 Experimental Biodegradable Lubricating Greases

Composition	MIL-G-10924F	BLG-1	BLG-2	BLG-3
Base Oil	PAO +Mineral	PAO +Mineral	PAO	Polyol ester +Diester + PAO
Biodegradation Improver, (Rapeseed Oil)	None	10 %	20 %	None
Li-Complex Thickener	15	15	15	15
Other Additives	10	10	10	10

* BLG - Biodegradable Lubricating Grease

2.1. LABORATORY BIODEGRADATION TEST

Biodegradation is a natural process caused by the action of microorganisms. In the presence of oxygen, nitrogen, phosphorous, and trace minerals, organic pollutants can support microbial growth and are converted into a series of oxidation products that generally conclude with carbon dioxide and water. The biodegradation test method adopted in this study follows the ASTM D5864, “Standard Test Method for Determining Aerobic Biodegradation of Lubricants and Their Components”. Recently, ASTM D-12 Subcommittee on Environmental Standard of Lubricants have developed this biodegradation test method based on the OECD Modified Sturn Test which closely simulates the wastewater biotreatment conditions [8]. This test method was originally designed to determine the degree of aerobic aquatic biodegradation of lubricants on exposure to an inoculum under laboratory conditions. In this test, the biodegradability of a lubricant is expressed as the percentage of maximum (theoretical) carbon conversion (or carbon dioxide generation) under well-controlled conditions for a period of 28 days.

Three experimental BLG products were tested according to the ASTM D5864 test method. A commercially available rapeseed based lithium grease and MIL-G-10924 grease were also utilizes as references. A summary of measured biodegradation results of five test samples is plotted in Figures 3. It is noted that these results were determined based on duplicate test results in order to increase reliability of test data and are expressed as percentage of the maximum (theoretical) carbon dioxide evolution of each test sample. Figure 3 shows that the biodegradability of 28 days ranges from 28% for MIL-G-10924F grease to 64% for BLG-3. The rapeseed-BLG provides a biodegradability of 48.9 %, while experimental BLG-1 and BLG-2 mark 46.5% and 45.3%, respectively. All BLG products exhibited a higher biodegradability than MIL-G-10924F grease and the BLG-3 provides the highest biodegradability among all the tested greases. It may be stated that greases from either natural source or synthetic ester derives are better biodegradable materials than petroleum-based products. Especially, the BLG-1 with 10% rapeseed oil gave 66 % improvement of biodegradability over the baseline grease, while BLG-2 with 20 % rapeseed oil improved the biodegradability by almost 59 %. Both results were very similar to the biodegradability of 100% rapeseed-BLG. It may generally be concluded that a small amount of biodegradability improver (rapeseed oil) which bacteria like to eat due to its natural ester flavor may substantially improve the biodegradation process of a lubricant. Also, Figure 3 demonstrates that the biodegradation process of rapeseed-BLG starts earlier than the other three greases and slows down after first week of test. The BLG-2 with 20% rapeseed oil provides a similar biodegradation profile to rapeseed-BLG, while the biodegradation behavior of BLG-1 with 10% rapeseed oil was similar to its base grease (MIL-G-10924F). Unlike the other types of greases, the synthetic ester based BLG-3 tends to steadily increase its biodegradability over 28 days. It appears that the biodegradability of greases highly depends on the types of base oils used in the grease formulation.

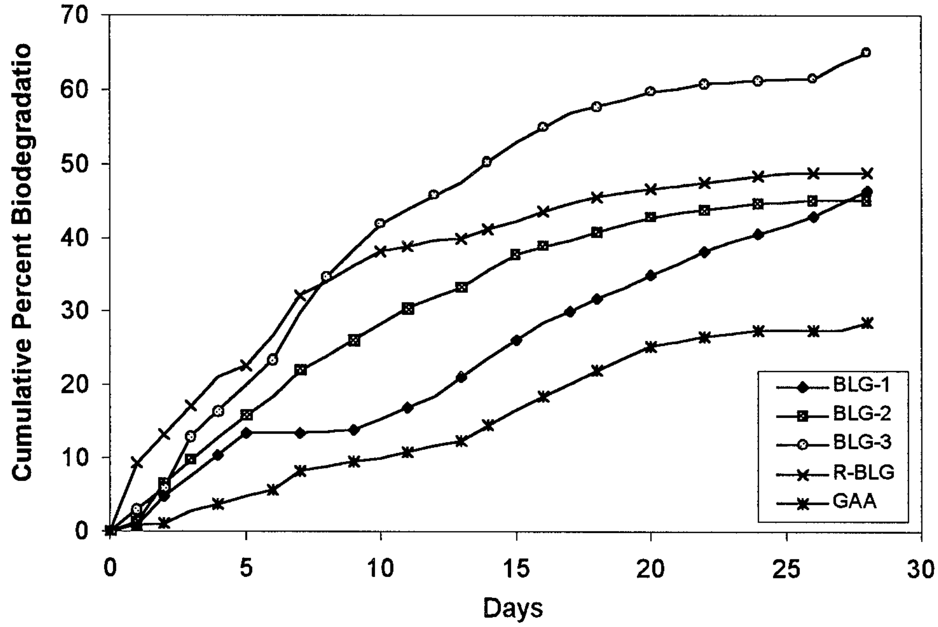

Figure 1. Biodegradation Profile of Lubricating Greases

2.2. PERFORMANCE CHARACTERSTICS

Military greases are currently used in various mechanical equipment systems designed for open lubrication system such as automobile wheel bearings, chassis systems, and gearboxes. As a plan, the experimental BLG products were also designed for use in the same mechanical applications. To evaluate the performance of biodegradable lubricating grease, the BLG-3 was selected based on its high biodegradability between the other interim products. A commercial vegetable-based grease (referred to as rapeseed-BLG) was also tested against the BLG target requirements. For comparison purposes, a product qualified under MIL-G-10924F.was also tested for the baseline study. These greases were evaluated according to the test protocol and the results are summarized in Table 2.

The most distinguishing property of grease is its consistency that is related to the hardness or softness of the grease. The consistency is rated by the penetration number. It is defined as the depth, in tenths of a millimeter, that a standard cone penetrates a sample of grease under prescribed conditions of weight, time and temperature. To ensure a uniform sample, a grease is worked 60 strokes using a grease worker before running the penetration test. The results are classified by grade ranging from 000 (very soft) to 6 (very hard) using National Lubricating Grease Institute (NLGI) grease classification system [10]. The test results showed that all samples rated as NLGI No. 2 grade that is designed as being a medium consistency. It indicated that the vegetable oils can also be used as base oil of greases like petroleum-based oils.

The mechanical stability of greases is usually evaluated by the work and water stability tests. In these tests, the stability of greases is determined based on the measurement of penetration changes in consistency due to the continuous application of shearing forces with and without water present. If a grease has a mechanical stability problem, it will usually appear normal before being subjected to service but will soften rapidly or harden upon working. Concurrently, it can lead to a lubrication failure within mechanical components. In these tests, Rapeseed-BLG had substantial water problem in stability, while BLG product did not show any abnormal behavior. It appeared that vegetable-based BLG products have some degree of mechanical stability problems under wet condition. The thermal stability of the lubricating greases is currently comprehensively evaluated using the results obtained from the dropping point and evaporation tests. The dropping point tends to measure the high temperature operability of greases and dependent on the type of thickener used in grease. A high dropping point grease usually provides better thermal stability for extension of bearing life at high temperatures. Rapeseed-based BLG showed a medium dropping point, while both BLG-3 and MIL-G-10924F grease had a high dropping point due to their lithium-complex thickener system. The evaporation loss at elevated temperatures also indicates the degree of the thermal stability of greases. Rapeseed-BLG had a higher evaporation loss than the other two greases due to its base oil. The BLG-3 product provides excellent thermal stability at this test temperature (180 °C).

Oxidation stability is another important property of biodegradable greases and is intended to predict their storage and service life. To evaluate oxidative life of biodegradable lubricating greases, oxidation tests were conducted using ASTM D5483, Pressure Differential Scanning Calorimeter (PDSC) method. This method was recently developed to assess oxidation stability of the lubricating greases by measuring the differential heat flow between sample and a reference thermocouple at various temperatures (155 , 180, 210 °C) under a pressure of 3.5 MPa. In this procedure, the degree of oxidation stability at a given temperature is determined by measurement of induction time. The PDSC test results showed that rapeseed-BLG had a lower induction time than those of the other two greases. This result also agreed with the results of the thermal stability tests.

Tribology (friction, wear, and lubrication) properties are one of the important operational parameters in conventional mechanical systems. Most lubricating greases often use anti-wear additives to improve their wear prevention properties. This property is usually evaluated by the ASTM D 2264 Four-Ball Wear Test. The test results indicated that biodegradable lubricating greases do not have any compatibility problem with conventional anti-wear additives. This result was confirmed by ASTM D 2596 Four Ball Extreme Pressure (EP) tests.

Excessive oil separation of greases often indicates grease degradation in service/storage period. To assess this physical property, a static oil separation test was conducted using the Federal Test Method 791.321[11]. The results did not indicate any abnormal oil separation from either biodegradable lubricating greases. To verify this result, dynamic oil separation tests were conducted using the modified ASTM D4534 Method, Oil Separation from Lubricating Grease by Centrifuging (Koppers Method). The results showed that both biodegradable lubricating greases were physically stable under centrifugal force and provided the same quality of performance observed with the MIL-G-10924F grease.

For corrosion protection property, both biodegradable lubricating greases did not exhibit any corrosion problem with copper metal. In the saltwater rust tests, Rapeseed-BLG gave medium corrosion spots on the test bearing surface, while the other two greases did not show any corrosion problem. This chemical property is one of the more important operational parameters for the overall military readiness.

The low temperature property of greases is also one of important operational parameters in mechanical systems. If grease becomes too hard under sub zero temperatures, mechanical systems such as bearings, can lose lubrication and require higher torque. This can result in fatigue failure of mechanical system. Currently, this property is measured using a mechanical torque tester that was simulated from an automobile wheel bearing system. For the evaluation, three grease samples were tested at - 54 °C, using the US Army Low Temperature Torque test procedure [12]. The results showed that vegetable-based grease have very limited low temperature capabilities, while synthetic-based grease provides excellent rheological properties. It appeared that vegetable-based grease (Rapeseed-BLG) contains a wide range of fatty acid esters and tends to crystallize into long-chained esters of fatty acids well above the measured pour point.

The high temperature service life of grease is an another important operational parameters in mechanical systems. This property usually defines the upper operational temperature in service. Currently, several functional test methods are available to measure grease high temperature life. Among them, ASTM D3537 Method, Life Performance Test of Lubricating Greases, is widely used in the grease industry and by users. This method comprehensively evaluates all individual physical properties of greases directly related to high temperature and shear, using a simulated front wheel bearing system and a dynamic laboratory bench-type test apparatus [13]. Table 3 showed that the Rapeseed-BLG provides only 30 hours of high temperature life due to its poor thermal and oxidation stability. Such a grease can not be used in high temperature applications such as wheel bearings. On the other hand, the experimental BLG-3 product provided excellent high temperature life and its performance is equivalent to that of MIL-G-10924F grease.

Table 2 Laboratory Performance Test Results for Biodegradable Greases

Test Method		Target Requirement	Rapeseed-BLG	BLG-3	MIL-G-10924F (GAA)
Dropping point, C	ASTM D2265	240, min	178	272	278
Worked penetration, 1/10 mm	ASTM D217	265-295	250	289	282
Work stability, 100,000 strokes	ASTM D217	-25 to 60	+61	+32	+18
Roll stability	ASTM D1831	-25 to 60	+22	+15	+9
Evaporation loss, %, 180 C, 1 hr	TGA	15, max	2.67	12.1	25.0
Oil separation, %	FED.791.321	10, max	2.5	2.79	1.6
Centrifuge, oil separation, 2hrs, 40 C, %	Mod, ASTM D4425	18, max	3.8	17.2	8.8
Four Ball EP, Load Wear Index (LWI), kgf	ASTM D2596	30, min	27	38.5	42.2
Four ball wear scar dia., mm	ASTM D2264	0.6, max	0.58	0.53	0.48
Copper corrosion	ASTM D4048	1b	1b	1a	1b
Water stability, 1/10 mm	Mod. ASTM D217	-25 to 60	+100	-8	+41
Saltwater corrosion, 1% NaCl	Mod, ASTM D1743	No corrosion	Medium corrosion	No corrosion	No corrosion
Low temperature torque, -54 C, N·m	Army method	Breakaway: 7 Running @5min:5, max	31.6(B) 4.63(R)	3.1(B) 1.5(R)	4.2(B) 2.2(R)
PDSC, min	ASTM D5483	10, min	14.3 (155 C)	23.6 (210 C)	12.9(210 C)
Elastomer compatibility, %	ASTM D4289	10, max	ND	ND	ND
Biodegradability, %	ASTM D5864	60, min	48.9	64	28
Toxicity	OECD	>1000	ND	ND	ND
Grease life, min, hr	ASTM D3527	100, min	30	100	130

ND: not determined

3. FIELD DEMONSTRATION PROGRAM

3.1. OBJECTIVE

The objectives of this field demonstration was to verify performance of a candidate BLG product as a potential replacement for Grease, Automotive and Artillery (GAA) in existing military equipment. Successful completion of this demonstration would result the current MIL-PRF-10924 grease being replaced with a non-toxic and biodegradable product.

3.2. SCOPE

The field demonstration encompassed two sites, Ft. Hood TX and Ft. Bliss TX, and focused on lubrication performance and the potential environmentally acceptability of candidate biodegradable GAA. The candidate BLG product was evaluated in a wide variety of military vehicles and construction equipment. The duration of this field test was designed for a year testing period. The final acceptance of the candidate BLG would be based on the field testing evaluation and resultant findings generated.

3.3. FIELD TESTING SAMPLE

A candidate grease (BLG 3) was selected as a field testing sample. This candidate BLG was fully evaluated under laboratory environments, and met all target requirements and current MIL-PRF-680 specification requirements. The physical/chemical properties of candidate BLG product are listed in Table 3. For the field demonstration, a 500 pounds of candidate BLG product was specially produced by a grease industry.

3.4 FIELD TESTING SITES AND PROCEDURE

A candidate BLG product was evaluated using the normal procedures utilized for GAA grease applications (i.e., wheel bearings). A total 10 vehicles and equipment was used for this demonstration at two different sites. Fort Bliss was a desert environment site, while Fort Hood was mild normal operational site. The test vehicles and equipment were selected based on availability and typical military application. The vehicles and equipment utilized in these demonstrations are listed in Table 2. In preparation of the demonstration, the existing GAA grease from wheel bearing system of the vehicles/equipment was completely removed, and maintenance personnel inspected the surface of these parts/components. The candidate BLG was then re-packed into the wheel bearings/CV joints according to maintenance procedures. The testing vehicles/ equipment must be operated a minimum for 10 hrs per week. During the tests, all performance should be observed, and periodically inspected. The test grease was sampled and evaluated its deterioration. As a part of this demonstration, a field biodegradation test was conducted to verify the environmental performance of BLG in compare with GAA.

• Ft. Bliss, TX evaluated a candidate BLG product using a contact truck, a Commercial Cargo Utility Vehicle (CUCV) Cargo truck, a loader backhoe, and three dump trucks at the 1st Combined Arms Battalion (CAS). These vehicles and equipment were used and maintained by personnel from the Intermediate Maintenance Division at Fort Bliss.

• Ft. Hood, TX also evaluated a candidate BLG product using two High Mobility Multipurpose Wheeled Vehicles (HMMWVs) and two 5-ton trucks at 602nd Maintenance Company, 13th COSCOM. These vehicles were used in routine military operations.

Table 3. Field demonstration vehicles and equipment

Code	Vehicle/equipment	Serial No/ Bumper No.	Location	Miles/Hours at change over
F-1	Contact Truck	IMD106	Ft. Bliss	23132
F-2	CUCV Cargo Truck	IMD 102	Ft. Bliss	93394
F-3	Loader Backhoe	342570	Ft. Bliss	444 hrs
F-4	Dump Truck A	1756DCAL2392	Ft. Bliss	46733
F-5	Dump Truck B	1751DCA12381	Ft. Bliss	66109
F-6	Dump Truck C	7DIF4FV53111	Ft. Bliss	42525
F-7	HMMWV-1	602-103	Ft. Hood	31078
F-8	HMMWV-2	603-200	Ft. Hood	39717
F-9	5-ton Truck	602-275	Ft. Hood	20712
F-10	5-ton Truck	602-273	Ft. Hood	21955

3.5. SCHEDULE

Milestone	Completion Date
Complete Test Plan and Preliminary Coordination	3Q FY 97
Identify Field Testing Sites and Complete Procurement of BLG product	4Q FY 97
Initiate Vehicle Field Demonstration at Ft. Bliss and Ft. Hood, TX	1Q FY 98
First Quarter Progress Review	2Q FY 98
Second Quarter Progress Review	3Q FY 98
Third Quarter Progress Review	4Q FY 98
Fourth Quarter Progress Review	1Q FY 99

Completed Vehicle Field Demonstration	2Q FY 99
Initiate Field Biodegradation Test at Ft. Hood	2Q FY 99
Completed Analysis of Field Sample from Vehicle Tests	3Q FY 99
Completed Field Biodegradation Test	4Q FY 99

3.6. DATA COLLECTION

All testing results and operator/user comments were recorded and reviewed on a quarterly basis. The following performance characteristics were closely monitored at each testing site.

• The lubrication performances of candidate grease was compared to existing GAA grease (e.g., overheating, starvation, excessive leakage, noise, etc.)

• Any material incompatibility was identified (e.g., softens plastics, elastomers, etc).

• Corrosion protection characteristics were evaluated (e.g., evidence of pitting, rust, discoloration, etc).

• Excessive wear on parts was noted (i.e., wear and scoring on the metal surface, etc.)

• Environmental assessment were determined (i.e., health and safety factors, operator acceptability, biodegradability, etc).

• Oxidation and water instability was observed at the field and the field sample was analyzed using laboratory equipment (i.e., PDSC, TGA, etc.)

• Biodegradability of candidate grease in service bearings was observed (i.e., biodegradation, fungy present, starvation, etc.)

3.7. POINT OF CONTACTS

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4. FIELD TEST RESULTS

A summary of the field test results is presented in Table 3. Data obtained for a candidate BLG-3 were generated from ten (10) military vehicles and construction equipment that were utilized in both Forts Bliss and Hood. Per the test plan, Fort Bliss evaluated the candidate grease using five vehicles (i.e., contact maintenance truck, cargo truck, dump trucks) and a construction equipment under desert environment, while Fort Hood evaluated a BLG-3 using four military vehicles (i.e., HMMWV, 5-ton trucks). These vehicles were used daily for their routine operation and military exercise. Code F-1 to F-6 represents the field test results obtained at Fort Bliss and Code F-7 to F-10 cover the test results from Fort Hood. The evaluation criteria used in this demonstration were their field performances (i.e., consistency, oxidation, wear and corrosion problem, oil separation, etc.) and environmental performance (i.e., biodegradability, toxicity). Quarterly, the candidate grease and parts (i.e., C.V. Joints, bearings) of the tested vehicles were inspected and the grease samples were collected from each vehicle for the laboratory chemical analysis (i.e., PDSC, TGA). Inspectors also recorded operated mileage, environmental temperatures, and road conditions. In this demonstration, the operated mileage from the test vehicles ranged from 343 miles to 5,446 miles. These mileages were normal peacetime military usage for a year. Representative photographs taken from the testing sites are provided in Appendix B.

Fort Bliss is located at desert environment and its annual temperature ranges from -20 C to 35 C. Under a desert environment, a candidate biodegradable grease was set up in the front wheel bearing systems of six military vehicles and equipment utilized in the Intermediate Maintenance Shop of the 1st Combined Arms Battalion (CAS). These wheel systems are normally lubricated with GAA. As a test plan, the vehicles and equipment were visually inspected quarterly the candidate grease and the wheel bearing systems, and a small amount of field sample were collected from wheel bearings for the laboratory evaluation. The inspection results showed that the candidate BLG-3 product did not give any abnormal behavior and clearly demonstrated the acceptable performance equivalent to the current military grease (MIL-G-10924). In addition, it did not show any biodegradation in the tested bearing systems during the field demonstration period. This indicates that BLG product can only be biodegraded under certain environmental conditions (i.e., ground soil or water). The laboratory test results obtained from field samples also clearly supported to these test results. The oxidation induction times (PDSC) generated from the BLG field sample showed some correlation to the vehicle operated mileage.

Fort hood also evaluated the BLG-3 product using four (4) military tactical vehicles. For the field demonstration, a BLG-3 was lubricated at Constant Velocity (C.V) Joints of HMMWV, front wheel bearings and chassis of other 5-ton vehicles. In a quarterly inspection period at Fort Hood, it was observed that oil washout problem was found on rear axial system of M931, 5-ton vehicle. It appeared that 80/90 differential oil used in the rear four axles had wash out the candidate BLG lubricated in the wheel bearings. In addition, a very severe oil leakage problem was observed at the real axial system. According to the military personnel from the 602nd maintenance Co/13th COSCOM, the cause of this problem has been identified as a main axle design problem of all 5-ton trucks. Specially, the rear axle seals of all 5-ton vehicles used in Fort Hood were heavily leaking. Because of this, the candidate grease was dissolved and washout from bearings. For the test, the rear bearings were re-lubricated with candidate BLG grease. Ft, Hood Science advisor has contact M931 truck program office and requested their action on this oil washout problem. However, any defective mechanical parts can not provide good lubrication system. This problem can eventually lead grease failure in wheel bearing systems. Except for this design problem, the candidate biodegradable grease provided an excellent lubrication performance in the tested vehicles.

In the environment assessment, the candidate BLG product is non-carcinogenic and does not contain any ingredients listed by EPCRA, CERCLA, and RCRA. Also, worker exposure is not regulated by OSHA. During the field demonstration, the candidate BLG product was handled by a normal maintenance procedure and did not give any skin and eye irritation on maintenance people. This grease is a low toxic product and met all toxicity regulations. However, this grease was not originally formulated as a food grade. No one complained that BLG product has a health problem as well as GAA does.

To assess biodegradability of the candidate BLG, a field biodegradation test was also set up at the bioremediation site of Fort Hood, TX. A candidate BLG-3 was tested using a modified Fort Hood's remediation procedure. For the reference purpose, the conventional MIL-G-10924 grease was also tested at Fort Hood. For the test, the samples were mixed with soil and then, plowed and tilled in order to increase homogeneity. No fertilizer and microbes were applied except for water. Soil samples have been obtained on monthly basis and analyzed for total carbon content using the EPA method 413.2, Spectrophotometric, Infrared for Total Hydrocarbon from Wastes. Results obtained from this field demonstration have clearly shown the rapid biodegradability of BLG-3 sample as compared to the extremely slow biodegradability of conventional MIL-G-10924 grease. The data obtained from the field biodegradation test were plotted in Figure 4 and demonstrated a good correlation to the laboratory biodegradation test used in this study.

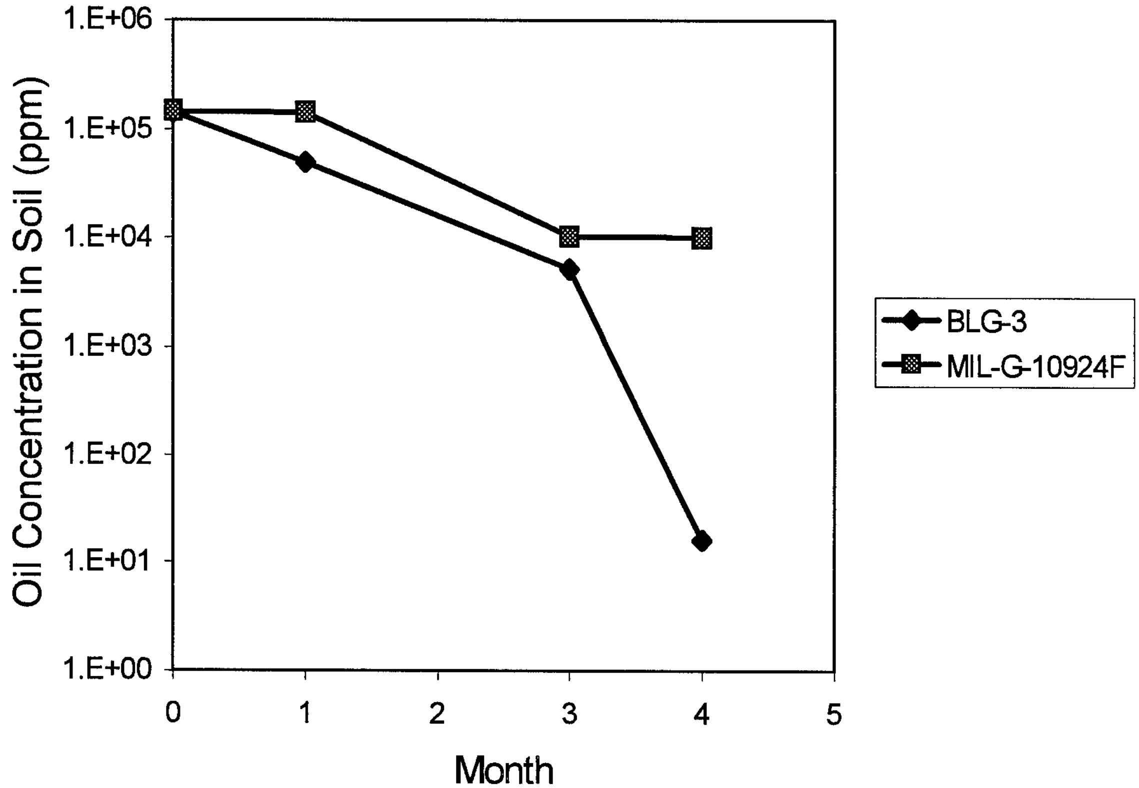

Figure 2. The Results of Field Biodegradation Test

Table 4. Results of Field Demonstration for Biodegradable Grease

CODE	IDENTIFICATION	INSPECTED PART	OPERATED MILEAGE	PDSC Minute	OIL content (%)	VISIUAL INSPECTION
F-1	Contact Truck	Front wheel bearings	5,446.2	27.4	78.5	. no consistency change . no wear and corrosion . color change to dark green . good grease condition
F-2	CUCV Cargo Truck	Front wheel bearings	2,712.8	46.3	78.6	. no grease deterioration . no oil separation . no biodegradation . good grease condition
F-3	Loader Backhoe	Front wheel bearings	134.7 hours	64.0	81.1	. no consistency change . no grease deterioration . color change to dark green . good grease condition
F-4	Dump Truck A	Front wheel bearings	916.9	37	79.1	. no oil separation . no consistency change . color change to dark green . good grease condition
F-5	Dump Truck B	Front wheel bearings	1843.3	59.1	80.7	. no grease deterioration . no biodegradation . no wear and corrosion . contamination with sand and water
F-6	Dump Truck C	Front wheel bearings	1337.5	72.5	80.7	. no consistency change . color change to dark green . no biodegradation . good grease condition
F-7	HMMWV-1	C.V. Joint systems (front-rear)	3044	50.6	76.4	. no consistency change . no color change . no corrosion . good grease condition
F-8	HMMWV-2	C.V. Joint systems (frond-rear)	2501	41.4	81.2	, no grease deterioration . no oil separation . no color change . good grease condition
F-9	5-ton Truck	Front-rear wheel bearings	343	173	83.8	. no consistency change . no biodegradation . no color change . good grease condition
F-10	5-ton Truck	Front rear wheel bearings	869	131	79.4	. no corrosion . no consistency change . no color change .good grease change
F-11	New BLG-3	-	0	324	87.7	. brown color

5. CONCLUSIONS

Biodegradable greases have been comprehensively studied in order to evaluate their biodegradability and lubrication performances against the conventional petroleum based-greases. Synthetic ester based BLG-3 formulated for this study provided excellent biodegradability and exceptional lubrication performance under extreme conditions. This grease was specially blended with three different types of biodegradable oils (i.e., polyol ester, diester, polyalphaolefin) in order to meet the requirements of a proposed military biodegradable grease specification. The field test results showed that BLG-3 product was compatible with conventional non-biodegradable synthetic oil-based grease and met all requirements of the designated specification that was designed for extreme environments. The field biodegradation test also showed that the candidate grease provide an excellent biodegradability over the conventional GAA grease. Also, the field test results were found to be a good agreement with the laboratory biodegradation test used in this study.

All field demonstrations were successfully completed and met original milestones and did not impact to the military mission of the unit in any way. The overall performance of candidate BLG product has been proved in the field demonstration and accepted by military users. Therefore, the BLG-3 grease can be considered as a next generation of military multipurpose grease.

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Appendix A. Biodegradation Test Method

A.1. Test Apparatus:

A schematic diagram of the experimental biodegradation test apparatus for ASTM D5864 test is shown in Figure 1. This test apparatus was a slightly modified for the study. It consists of four separate units: the air supply/carbon-dioxide scrubbing system, the incubation/biodegradation batch reactor, a carbon-dioxide collector, and a titrator. Both the carbon dioxide scrubbing and the biodegradation units utilize Erlenmeyer flasks. To eliminate other carbon sources, except the test lubricant, CO_2-free air is needed for the biodegradation test. A laboratory compressed air supply was attached directly to the carbon dioxide scrubbing system to produce CO_2-free air. The scrubbing system uses cascade flasks: two flasks containing 10 M potassium hydroxide (KOH) solution and two flasks containing 0.025 M barium hydroxide $Ba(OH)_2$ solution. To ensure the desired aerobic environment, the test solution containing the test lubricant was fully agitated using a variable speed magnetic stirrer. In order to conduct multiple tests for performance comparison, ten separated identical biodegradation batch reactors were connected as seen in Figure 1. Each reactor can be independently and flexibly operated for aquatic biodegradation tests.

A.2.Test Procedure

Prior to the test, the test solutions were prepared according to the road diagram shown in Figure 2. Initially, five stock solutions for the test medium were prepared: ammonium sulfate solution (40 g/L), calcium chloride solution (27.5 g/L), ferric chloride solution (0.25 g/L), magnesium sulfate solution (22.5 g/L), and phosphate buffer (made of 8.5 g potassium dihydrogen, 21.7 g potassium monohydrogen phosphate, 33.4 g sodium monohydrogen phosphate, and 1.7 g ammonium chloride) It should be noted that these solutions do not contain any carbon material in order to avoid an extra source of carbon dioxide production. For a positive control and a fair comparison, a rapeseed oil was used as the reference sample. This oil has been identified to be a high biodegradable material. To determine the theoretical CO_2 evolution, the initial carbon content of the test lubricants was analyzed using the LECO CHNS-932 Elemental Tester [9]. The sewage inoculum (i.e., bacteria, yeast, fungi) was carefully prepared from the mixed liquor (approximately 1 liter) of activated sludge provided by a local wastewater treatment plant. This sewage inoculum was freshly collected timely from the biotreatment processing pool of the plant. It was fresh and contained the proper microorganisms for treating regular wastewater. In our laboratory, the total number of bacteria and fungi were observed and counted using the Easicult Bacteria Counting Kit. This test kit is commonly used for measuring the growth of bacteria in an industrial process, such as cutting fluids, etc. To avoid carry-over of sludge solids which might interfere with the measurement of CO_2 production, the sewage inoculum was homogenized by a blender and aerated until ready for use.

A total of ten test flasks were used for the biodegradation tests. Six flasks were designed for the test samples, two flasks as positive controls (baseline reference sample: rapeseed oil), and the remaining two flasks as blank controls. To prepare a one-percent inoculum solution, 2,470 mL of distilled water was added to each 4 L Erlenmeyer flask. Then, the following stock solutions (concentration explained earlier) were added to the test flasks: 3 mL of ammonium sulfate, magnesium sulfate, and calcium chloride; 30 mL of phosphate buffer; and 12 mL of ferric chloride stock solution. After all stock solutions were mixed and diluted in the 4 L Erlenmeyer flasks, 30 mL of the activated sludge inoculum mentioned earlier was added to the test solution.

To purge the CO_2 gas that might have migrated from the room air of the laboratory during the inoculum solution preparation period, the test flasks were aerated with CO_2-free air for 24 hours. Then, three CO_2 collectors filled with 100 mL of 0.0125 M $Ba(OH)_2$ solution were connected in series to the downstream of each 4 L Erlenmeyer flask. Before adding the test (or reference) lubricant samples, the pH values of the test solutions were all adjusted to 6.5 - 7.5 using HCl or NaOH solutions. About 80 mg of the test lubricant sample was added into each of the six test flasks. Rapeseed oil was also added into the duplicate positive control flasks. It is noted that the duplicate blank control flasks were free of test lubricants. Then, the distilled water was added to maintain a

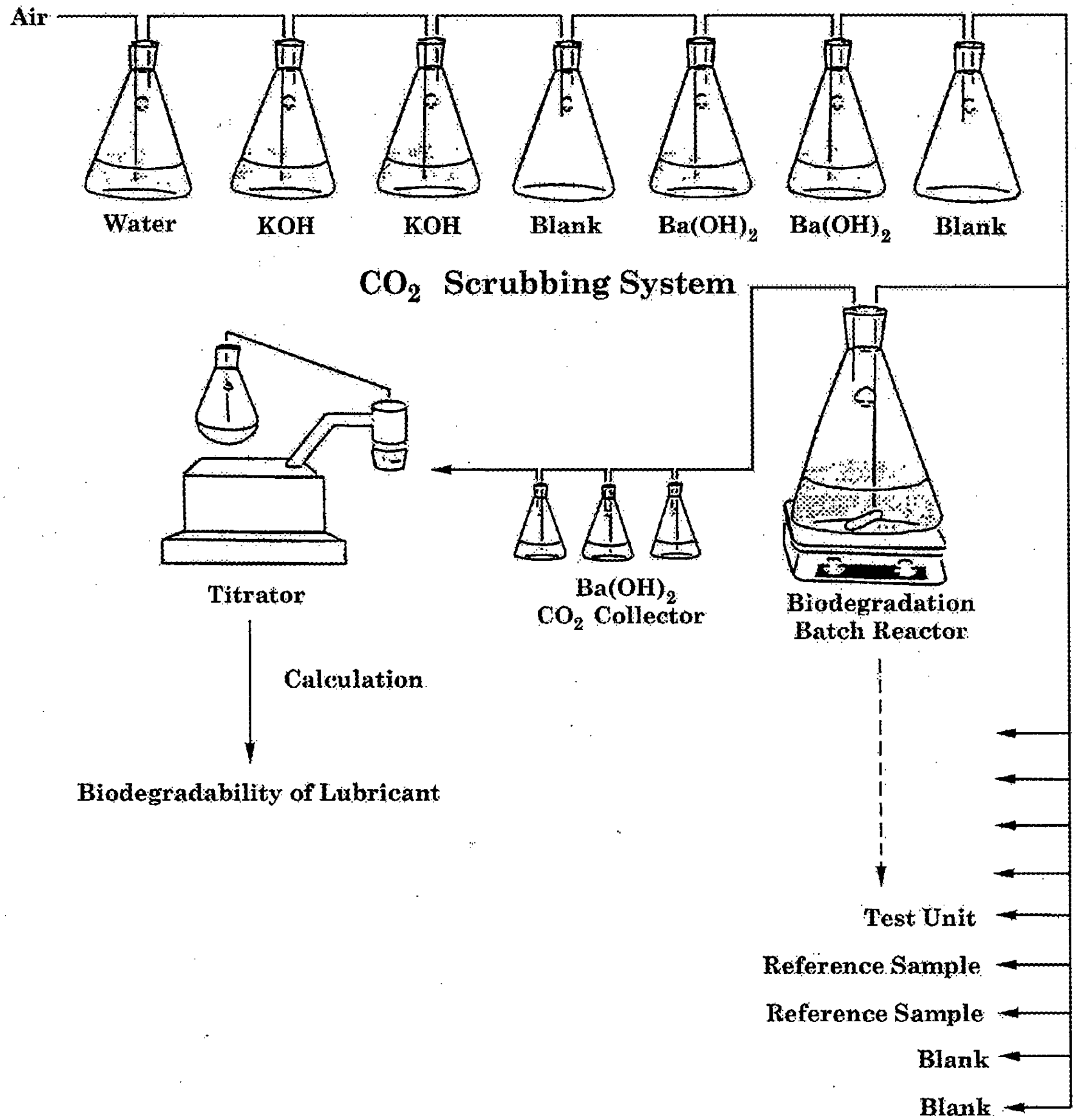


Figure A-1. Schematic Diagram of Biodegradation Test Apparatus

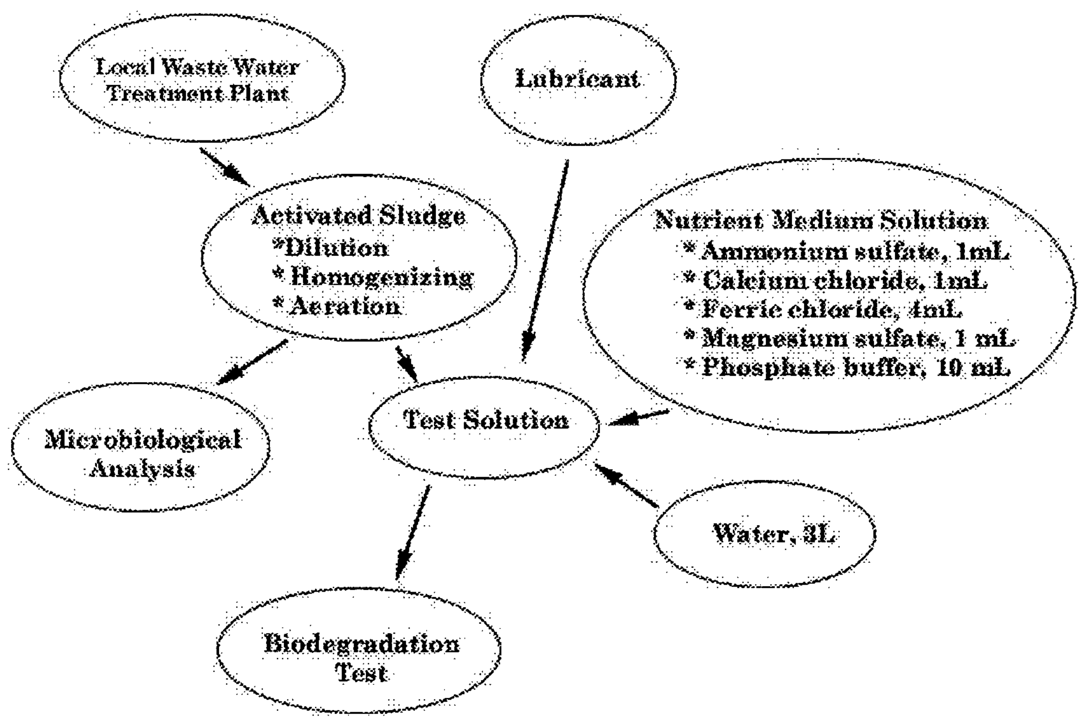


Figure A-2. Preparation of Test Solution

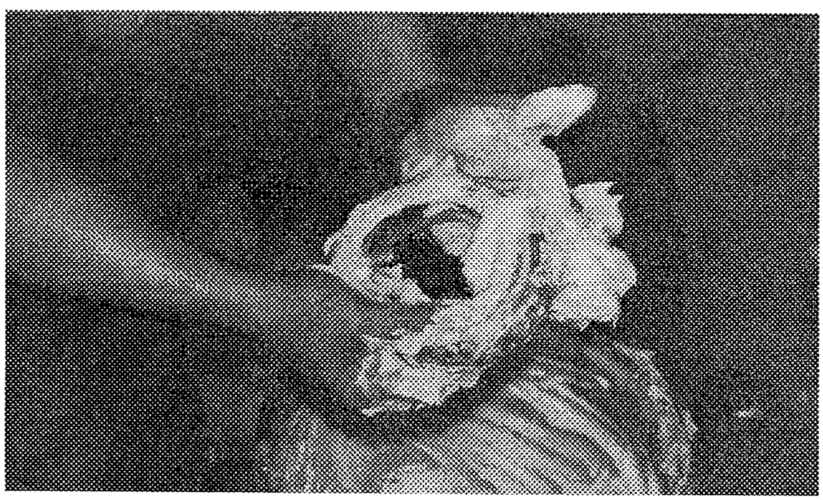
final volume of 3 liters in each 4 L flask. All test flasks were tightly stoppered and maintained at 20 - 25 C. The magnetic stirrer was kept at approximately 200 rpm. The volumetric flow rate of CO_2-free air to each test flask was maintained at 50 to 100 mL/min. It should be noted that each experiment included duplicate control flasks and duplicates of each test lubricant sample. During the test, the test room was kept in complete darkness. This measure was necessary to prevent photo degradation of the test substance and the growth of photosynthesis bacteria and algae.

To measure carbon dioxide production during a predetermined test period, the CO_2 collector nearest the 4 L Erlenmeyer flask was removed for titration and calculations. The remaining two collectors were moved up one place closer to the 4 L Erlenmeyer flask and a new collector filled with 100 mL of fresh 0.0125 M $Ba(OH)_2$ was placed at the far end of the series. Titration was performed every day for the first 10 days and then every other day for the remaining 18 days or until a plateau of CO_2 evolution was reached. The end point used for automatic titration was set at pH 7. Once the CO_2 evolution has reached a plateau, the pH of the test solutions were measured and added 1 mL of HCL into the test solutions to decompose the inorganic carbonate and to release trapped CO_2 for a final titration. Data obtained from the titration were converted to the amount of CO_2 production using an equation specified by the method.

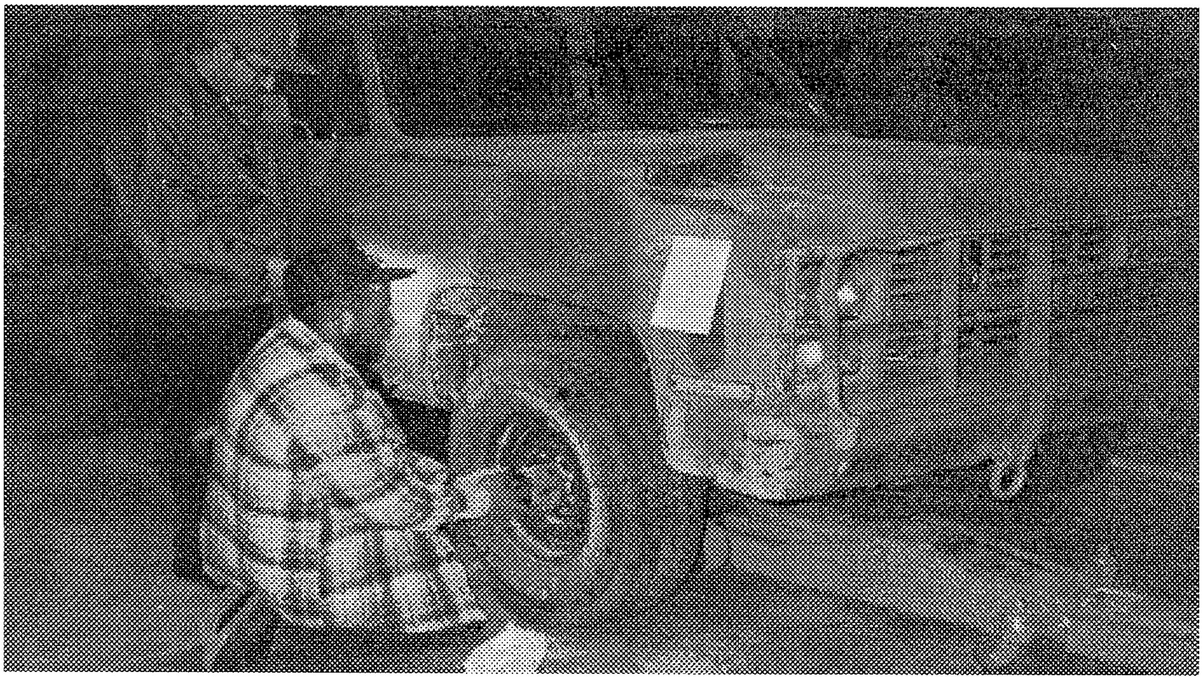
Appendix B

B.1 Fort Bliss Field Demonstration

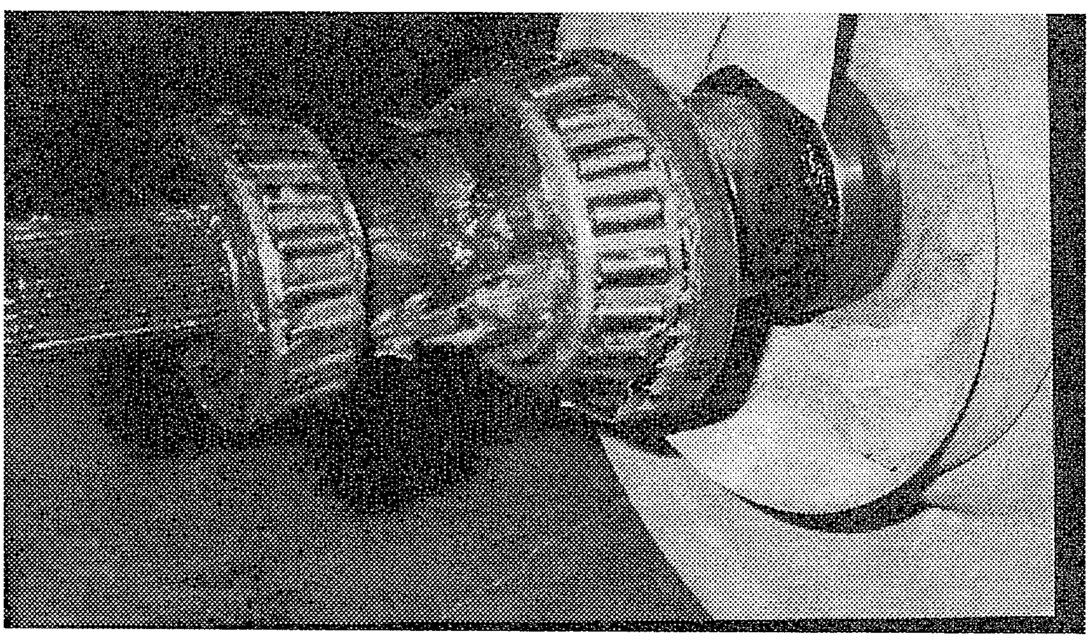
Contact Truck



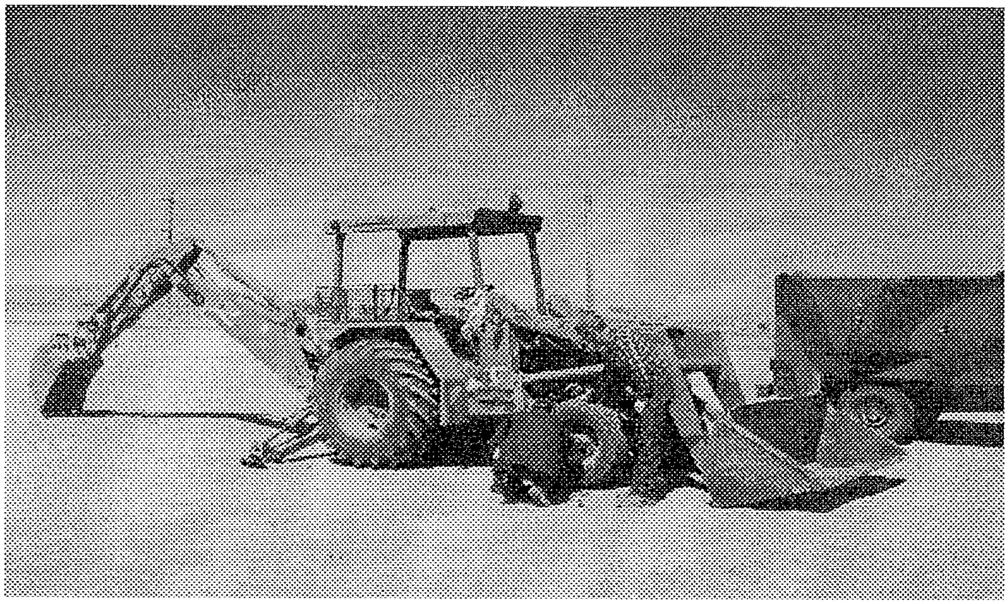
Grease Packing in a Wheel Bearing



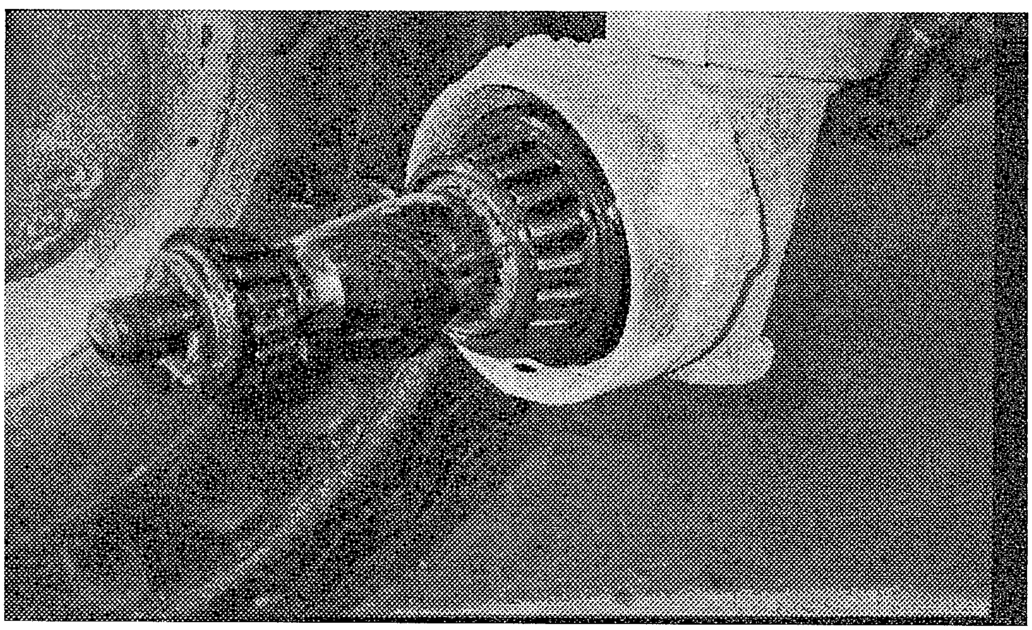
Installation of a Wheel Bearing lubricated with BLG



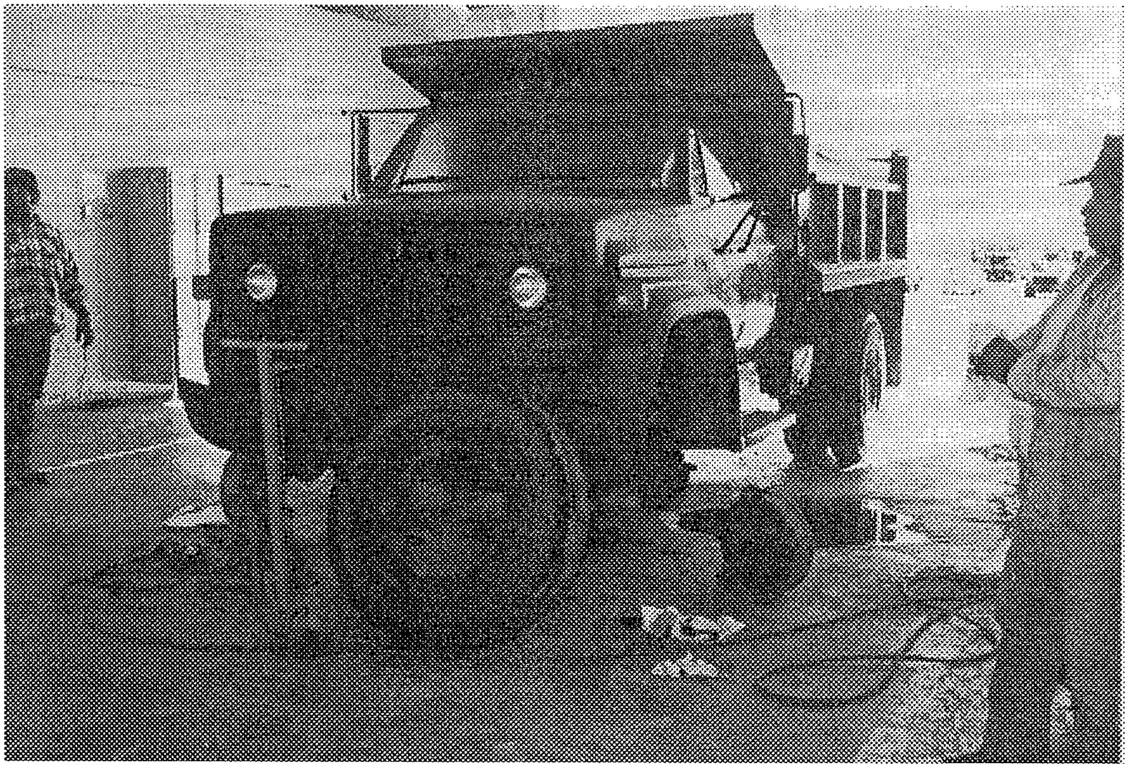
Quarterly Grease Inspection



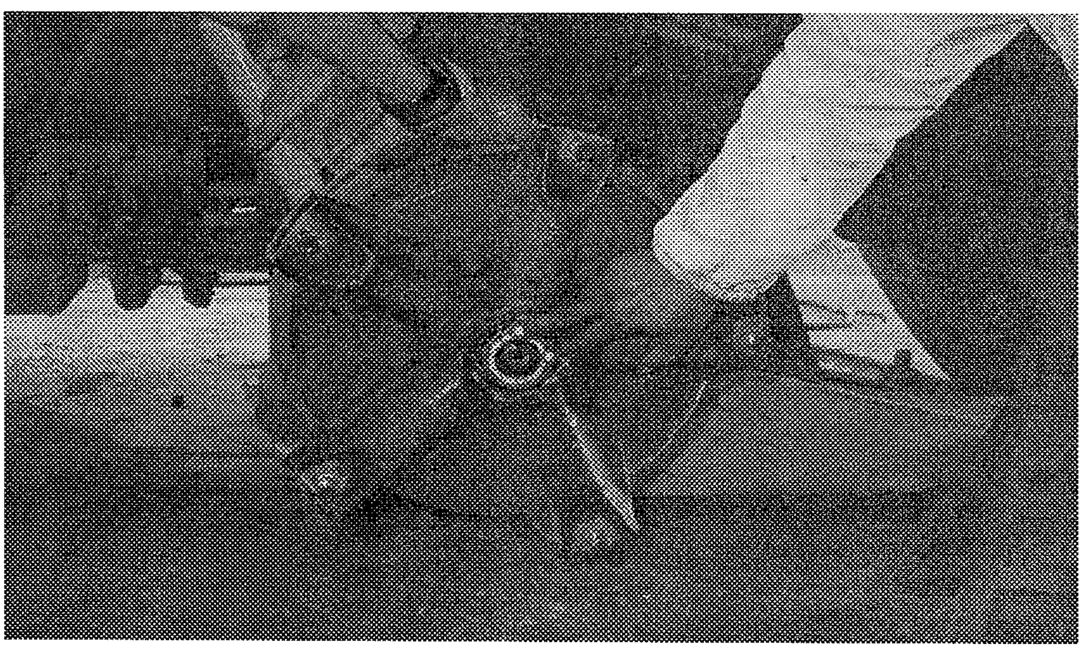
Loader Backhoe Equipment



Wheel Bearing System with BLG



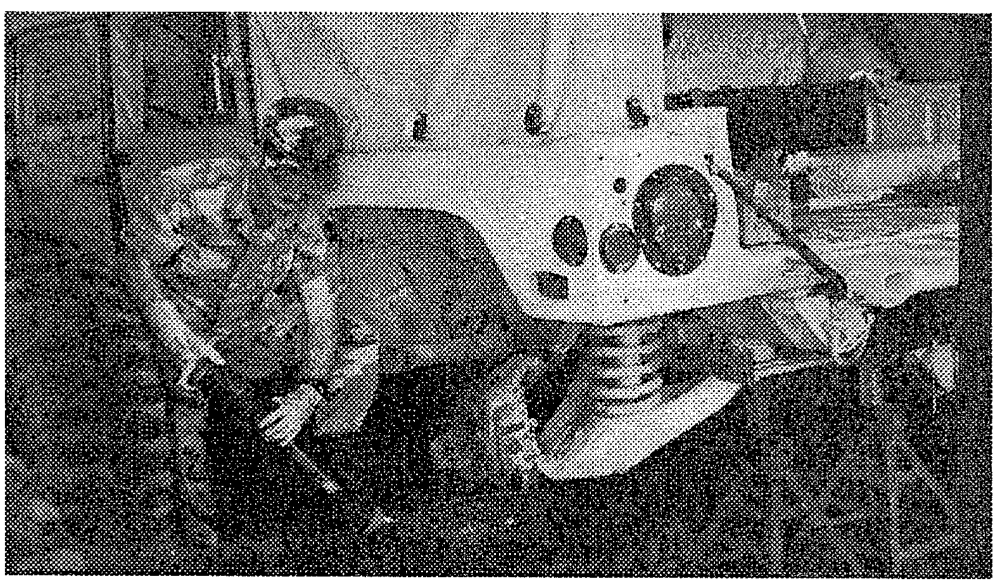
Dump Truck



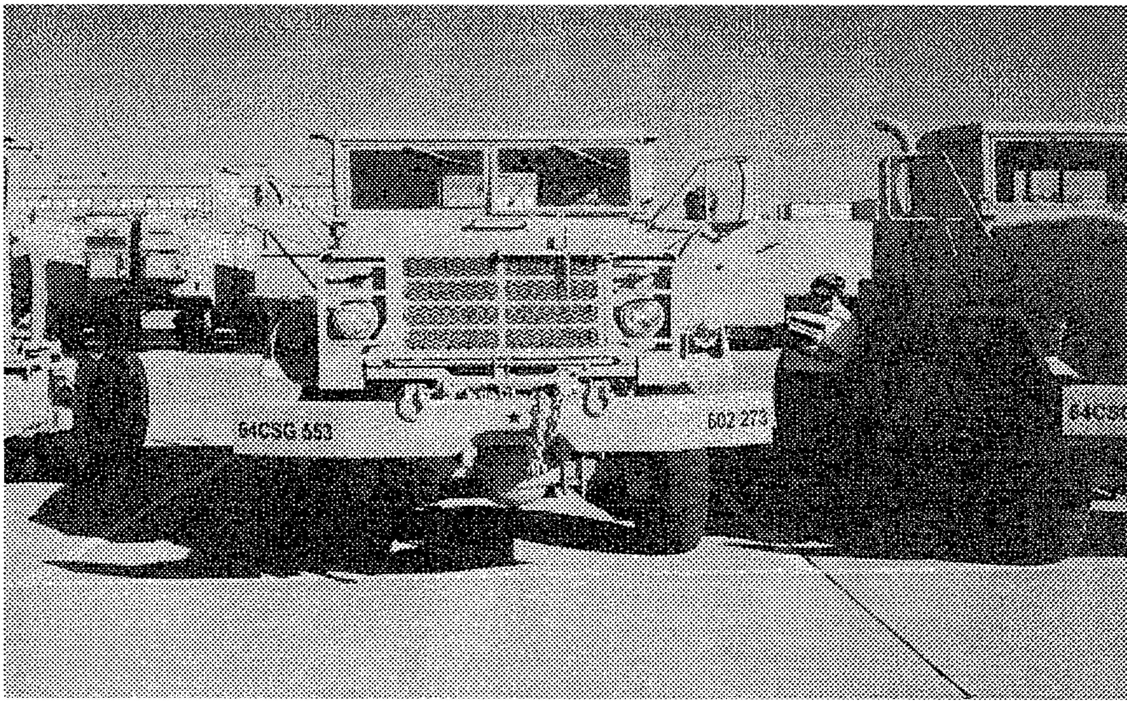
Demonstration of Wheel Bearing Inspection

B.2 Fort Hood Field Demonstration

HMMWV



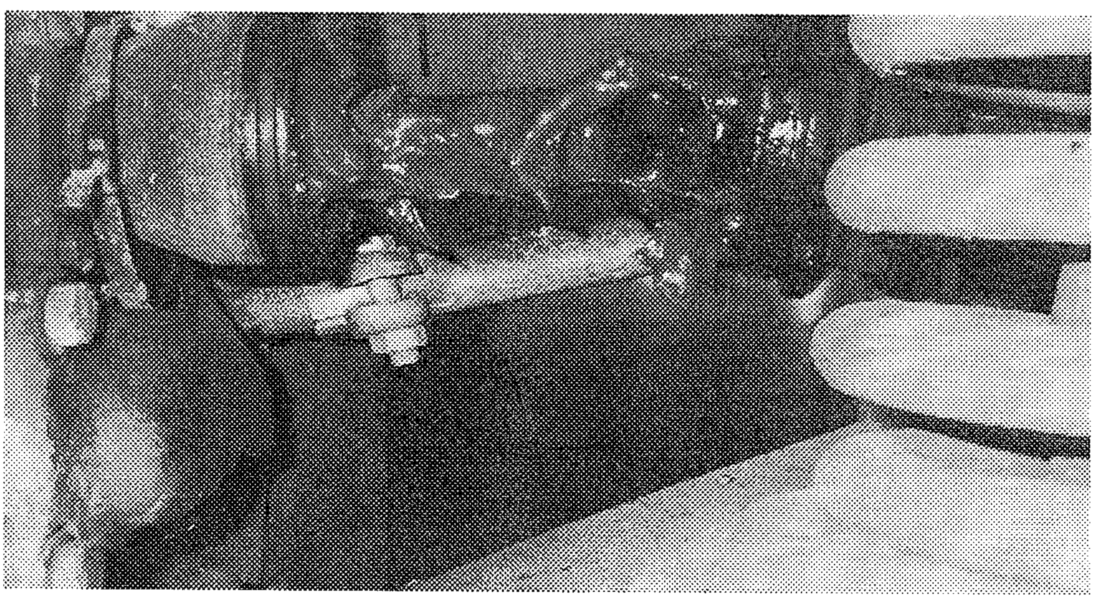
Quarterly Inspection of BLG in C.V. Joint



5-Ton Truck

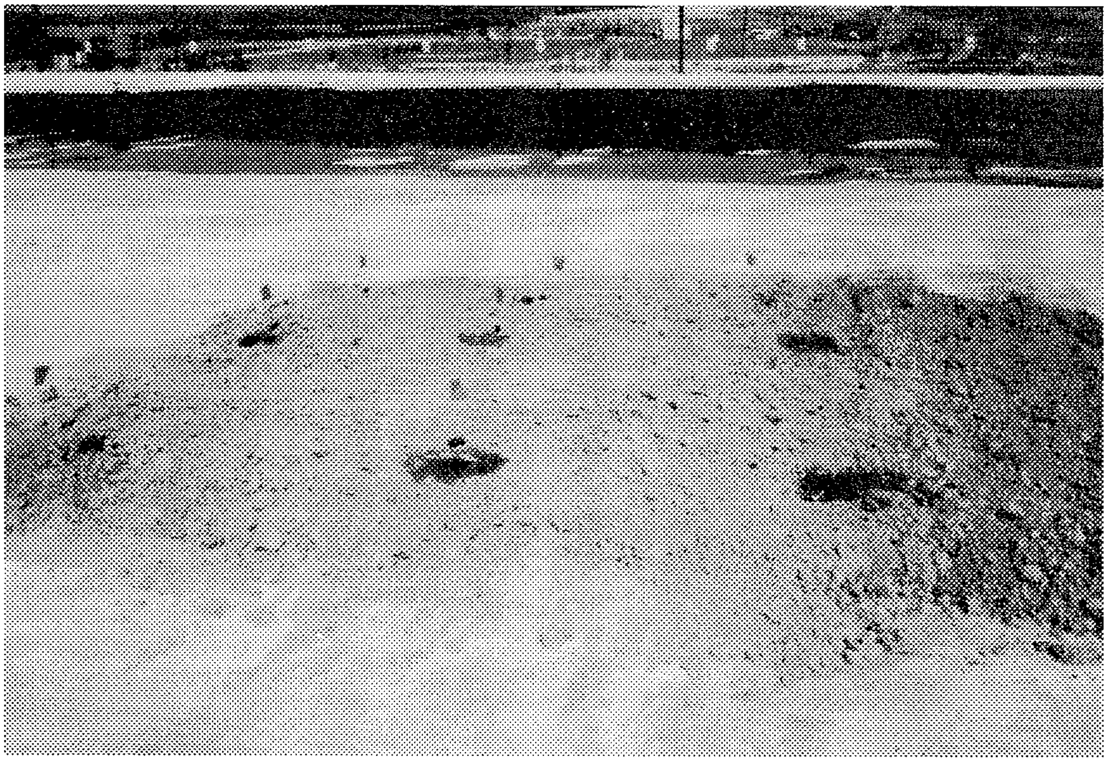
Removal of Wheel Bearing for Quarterly Inspection

Grease Inspection on 5-Ton Truck



C.V. Joint System in HMMWV

B.3. FIELD BIODEGRADATION TEST



Field Biodegradation Test for BLG at Fort Hood Remediation Site

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5880 CHANNEL VIEW BLVD
JACKSONVILLE FL 32226-3404

CDR
MARINE CORPS LOGISTICS BA
ATTN: CODE 837 1
814 RADFORD BLVD
ALBANY GA 31704-1128

CDR 1
FMFPAC G4
BOX 64118
CAMP H M SMITH
HI 96861-4118

CDR
NAVAL RESCH LABORATORY
ATTN: CODE 6176 (R. Mowery) 1
4555 OVERLOOK AVENUE, SW
WASHINGTON, DC 20032

CDR 1
2ND MARINE DIV 1
PSC BOX 20090
CAMP LEJEUNNE NC 28542-0090

Department of the Air Force

HQ USAF/LGTV
ATTN: VEH EQUIP/FACILITY 1
1030 AIR FORCE PENTAGON
WASHINGTON DC 20330-1030

AIR FORCE WRIGHT LAB 1
ATTN: W UPOS 1
WUPOSL
1790 LOOP RD N
WRIGHT PATTERSON AFB
OH 45433-7103

AIR FORCE WRIGHT LAB
ATTN: WUMLBT 1
2941 P ST STE 1
WRIGHT PATTERSON AFB
OH 45433-7750

SA ALC/SFT
1014 BILLY MITCHELL BLVD STE 1 1
KELLY AFB TX 78241-5603

SA ALC/LDPG
ATTN: D ELLIOTT
KELLY AFB TX 78241-6439

WR ALC/LVRS
225 OCMULGEE CT 1
ROBINS AFB
GA 31098-1647

AIR FORCE WRIGHT LAB
ATTN: WL/MLSE
2179 12TH ST STE 1
WRIGHT PATTERSON AFB
OH 45433-7718

AIR FORCE MEEP MGMT OFC 1
OL ZC AFMC LSO/LOT PM
201 BISCAYNE DR
BLDG 613 STE 2
ENGLIN AFB FL 32542-5303

Other Federal Agencies

NASA
LEWIS RESEARCH CENTER 1
CLEVELAND OH 44135

EPA
AIR POLLUTION CONTROL 1
2565 PLYMOUTH RD
ANN ARBOR MI 48105

RAYMOND P. ANDERSON, PH.D., MANAGER
FUELS & ENGINE. TESTING 1
BDM-OKLAHOMA, INC
220 N. VIRGINIA
BARTLESVILLE OK 74003

DOT 1
FAA
AWS 110
800 INDEPENDENCE AVE SW
WASHINGTON DC 20590

USDA
USDA-CSREES (Ms. Bailey) 1
AG BOX 2220
WASHINGTON, DC 20250-2220fd0